CONTRIBUTION TO CORROSION FATIGUE CRACK INITIATION MODELING

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ABSTRACT

The corrosion current noise time history contributes significantly to the knowledge of modeling the corrosion fatigue crack initiation for stainless steel under passive electrochemical conditions. The paper deals with the experimental analysis of the effect of the number of cycles on the corrosion current noise measured during corrosion fatigue crack initiation tests of 13%Cr6%Ni steel specimens submerged in the 0,01% NaCl water solution. The experiment instrumentation is described. Both the frequency of corrosion current events and the decay of the corrosion current magnitude when increasing number of loading cycles are discussed with relation to the theoretical model of corrosion fatigue crack initiation. The acoustic emission activity close to corrosion current events is discussed, too. A working hypothesis of passive film damage mechanism is postulated.

KEYWORDS

Corrosion fatigue crack initiation, stainless steel, modeling, corrosion current noise, acoustic emission

INTRODUCTION

Stainless steels containing more than 13% Cr are usually used in corrosive environments with the steel surface in a passive state. The characteristic of a passive corrosion behavior is the existence of a very thin oxide layer on the metal surface, which decreases the corrosion rate by five up to seven orders of magnitude compared to active dissolution. An effective corrosion reaction is possible only if this passive layer is at least partially destroyed. In the case of corrosion fatigue, repeated stress leads to damages of the protective passive film [1], [2].

The corrosion fatigue crack initiation behavior under passive corrosion is controlled by repassivation kinetics as well as by critical crack depth derived using fracture mechanics [2]. Daeubler et al. [3] developed a model of corrosion fatigue crack initiation under passive electrochemical conditions. The model is based upon the existence of a stable passive layer on the metal surface, which causes the metal not to be subjected to active dissolution except at slip step, when the metal is attacked by surrounding corrosion medium. Dissolution - repassivation process (event) is repetitively initiated and eventually penetrates along a slip step and into the metal matrix. For each dissolution - repassivation event, the penetration depth along a slip step equivalent to the amount of dissolved metal can be calculated, which is proportional to the area under the corrosion current decay curve. A good agreement of the experiment with model's data is attained for cycle asymmetry R = 0, for number of cycles less that 10^6, approximately. The corrosion fatigue behavior predicted by the model is independent on both the number of cycles higher then 10^6 and cycle asymmetry. However, the following facts has become known:

- Considerable drop of corrosion fatigue S-N curve, which can be expressed between 10^6 and 10^9 cycles by a factor of 0.5 to 0.3 [4], [5].
- Abnormal behavior of corrosion fatigue susceptibility to loading cycle asymmetry [6], [7].

Consequently, there has been prepared a research project focused on modeling of the corrosion fatigue crack initiation for stainless steel under passive electrochemical conditions. Particularly, supermartensitic steel [8] exposed to neutral aqueous solutions of chlorides will be considered. The model will be obtained for a number of loading cycles higher than $N > 10^6$ and for a set of loading cycle asymmetry values in the range $-1 < R < 0.6$.

The realistic events of dissolution - repassivation distribution in time form an important base for solving this project task. The Daeubler attempts to measure the current transients by in situ method (such as that of Pyle and co-workers [9]) were unsuccessful primarily because fatigue loads applied in his work [3] were very small, resulting in currents which were too low to be conveniently measured.

The formation of the mechanical damage releases energy in the form of an acoustic wave, which propagates through the specimen and may be detected using piezoelectric sensor, attached to the specimen. Some difficulties arise from the low energy emission associated with corrosion high fatigue crack initiation relative to other acoustic emission sources present during such testing [17].

Non-standard measurement equipment with both software and methodology for in situ measurement of corrosion current noise (CCN) as well as acoustic emission (AE) during corrosion fatigue tests has been developed in cooperation with DAKEL Company, ZD Rpety, Czech Republic [17].

The presented paper deals with the methodology of both CCN and AE signal measurements carried out during corrosion fatigue tests in order to gain knowledge of corrosion fatigue crack initiation processes.

EXPERIMENTAL

Experiments were conducted on the cast steel 13%Cr6%Ni, for which an extensive understanding of microstructure behavior is available [10], [11]. Samples were obtained from an air quenched (990° C) and twice tempered (580° C, 570 – 580° C) large casting. The chemical composition and mechanical properties are listed in Table1.

Hourglass-shaped specimens (with midsection diameter of 6 mm and with notch coefficient value of 1.1) were machined by grinding (Ra = 0,8 μm). Before testing, specimen surfaces were underwater polished by hand using abrasive paper (400 grit). The size of surface

Table 1.

	$R_p0,2$ [MPa]		R_m [MPa]		Elongation [%]		Contraction [%]		KV[J]
13%Cr6%Ni	682		873		16		50		85
	C [%]	Mn[%]	Si [%]	P [%]	S [%]	Cr [%]	Ni [%]	Mo[%]	PI
	0.05	0.69	0.35	0.014	0.019	13.05	6.16	0.44	14.5
13%Cr4%Ni	C [%]	Mn[%]	Si [%]	P [%]	S [%]	Cr [%]	Ni [%]	Mo[%]	PI
	0.018	0.61	0.64	0.018	0.003	12.89	4.37	0.53	14.6

microdefects were limited by 0,05 mm. Fatigue tests were conducted at room temperature, under free corrosion conditions, in a fatigue test resonance - type machine under force push-pull loading at a frequency of 130Hz. The load ratio $R = \sigma_{min}/\sigma_{max}$ ranged from 0,6 to 0,7. The experiment was focused on high cycle fatigue with numbers of cycles to failure $N_F > 10^6$.

The corrosion vessel was made of Plexiglas and it was sealed using non-conducting, refractory material. All corrosion fatigue tests were performed in a 100mg NaCl/1litre distilled water solution with air excess. The corrosion current noise (CCN) [12] representing the rate at which the dissolution - repassivation events occur was continuously recorded throughout the fatigue test. The CCN was measured using a 13%Cr4%Ni stainless steel electrode having the chemical composition listed in Table 1. The value of pitting index PI = Cr + 3,3Mo (weight %) is listed in Table 1, too. Both the specimen and the electrode were masked off above the water level using a refractory material (see above). Corrosion system potential vs. saturated calomel electrode (SCE), as well as solution temperature (Pt - thermometer) were monitored, too.

Resonance type acoustic emission sensors (DAKEL, type FTCCN02) were inserted between ends of the specimens and specimen grips using a viscosity couplant. Detected signals were transferred from the sensor through a preamplifier (43 dB) to a PC operated AE detector unit. For the testing undertaken in this work the full gain was set at 120 dB with a detected sensor signal threshold level of 1,7 μV. Using the acoustic wave velocity and the difference between the arrival time of AE signals for a particular AE event (microcrack, friction etc.) at each of the sensors, the location of the AE event could be determined.

All monitoring/recording of electrochemical quantity was performed either using SCE-temperature meter or CCN meter (type DAKEL CCN01). The modified measuring system of acoustic emission (type DAKEL XEDO05 with programs DaeMon for monitoring and DaeShow for result evaluation) was used. Input impedance were 500 Ω for the CCN input and 100 MΩ for the SCE input. Sampling rate frequencies were 20 Hz for CCN and 0,1 Hz (usually) for SCE and 2 MHz for AE.

Prior to the corrosion fatigue test, specimens were exposed to prefatigue test phase (in water solution) with a fraction of required fatigue test amplitude. Using this procedure, the stable passivity of specimen's surface was established.

RESULTS

Prior to fatigue tests, the system was experimentally verified for CCN/SCE measurability. For this purpose, the protective film of oxides was damaged using a sharp - edged glass piece. For CCN/SCE time records valid for damaged protective film of specimen surface, see Fig. 1.

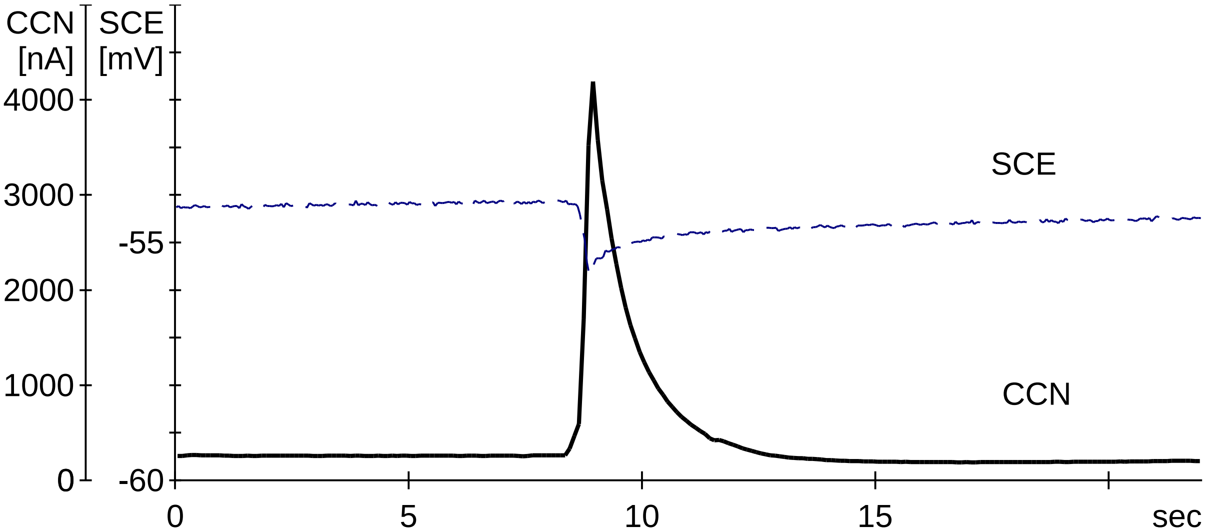

Fig. 1

The prefatigue test phase passed into the fatigue test procedure by a slow increase of the load cycle amplitude. The increase of the stress results in both an increase of CCN mean value and

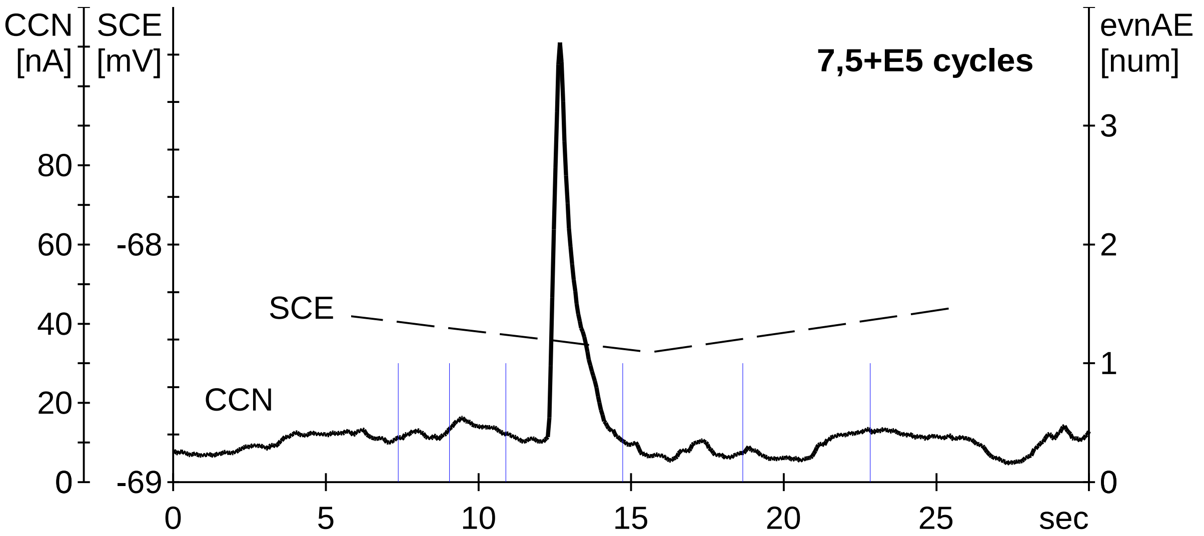

Fig. 2

a light decrease of the SCE. Usually, neither CCN transients nor AE events are registered. The CCN measurement during the corrosion fatigue test comprises slow accidental oscillations and saw-shaped transients with variable start/end steepnesses. Occurrence of the CCN transients correlates with minor SCE changes. Registered transient show the following features (see Fig. 2):

- Steep increase of CCN to the maximum value followed by a slower decline down to the original value.
- Steep decline of SCE down to the minimum value followed by a slow increase.
- Maximum CCN value coincides with the minimum SCE value.

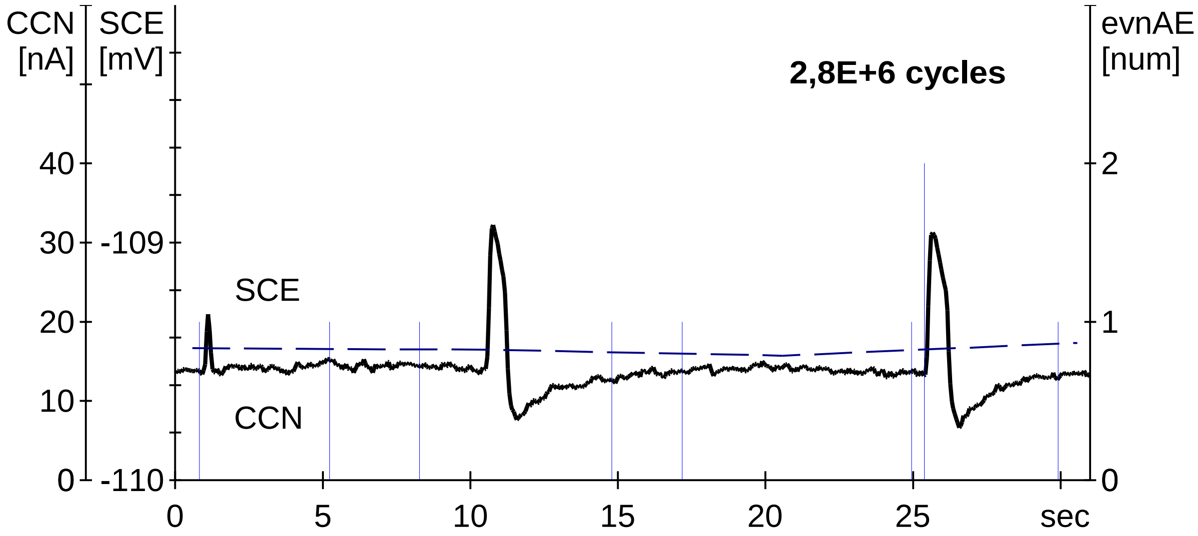

Fig 3

Acoustic emission events occur before CCN transients and close to the end of the transients, see Fig. 2, Fig. 3 and Fig. 4. The acoustic emission activity is lower during the CCN transients.

With increasing size of fatigue cracks, the CCN course pattern as well as magnitude show some changes. In the first third of the fatigue life, the specimen shows rarely CCN transients with minimal SCE variations – see Fig. 2. Rate of CCN transients increases gradually and the SCE values show lasting declination trend – see Fig. 3.

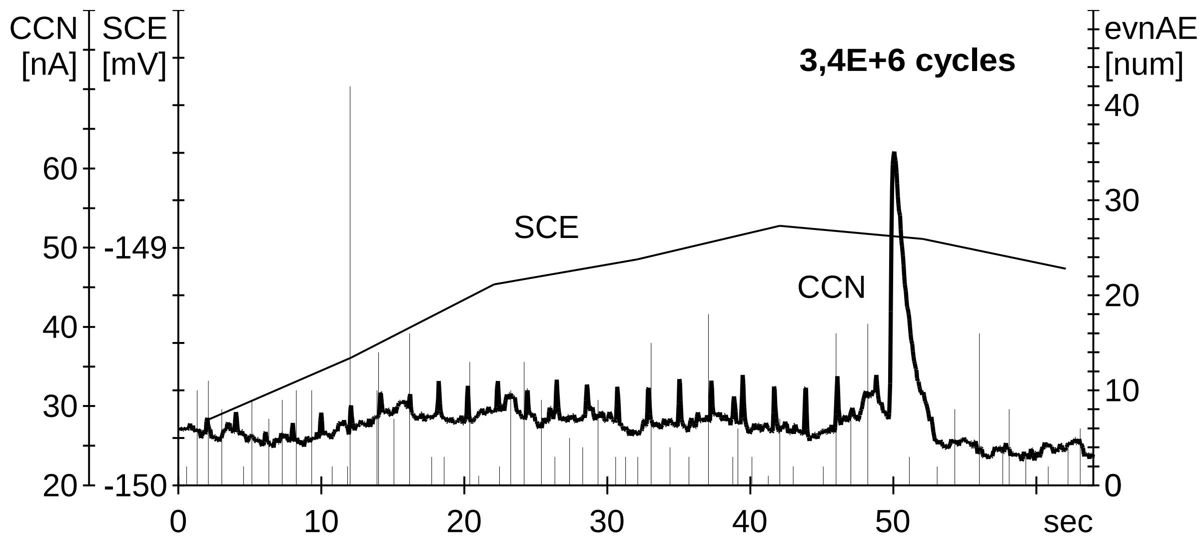

Fig 4

Prior to the end of the test, both the CCN transient rate and the AE events number increases together with a major decrease of the SCE – see Fig. 3 and Fig. 4.

DISCUSSION

Behavior function of CCN/SCE connected with the occurrence of CCN transients (see Fig. 2) is similar to the CCN/SCE function registered at trial protective film damage (using sharp piece of glass, see Fig. 1). Thus, CCN transient reflects the dissolution - repassivation process taking place after the passive film rupture. The CCN transient represents a corrosion current decay curve, whose parameters will be used as parameters for the corrosion fatigue crack initiation model.

The assumption adopted by Mueller and Daeubler in their model with respect to the course of the corrosion current noise during the dissolution – repassivation events appears to be valid. According to the Daeubler model, events need not appear at every cycle. The increase of the CCN transient rate with time complies with the theoretical consideration [13] and it may be

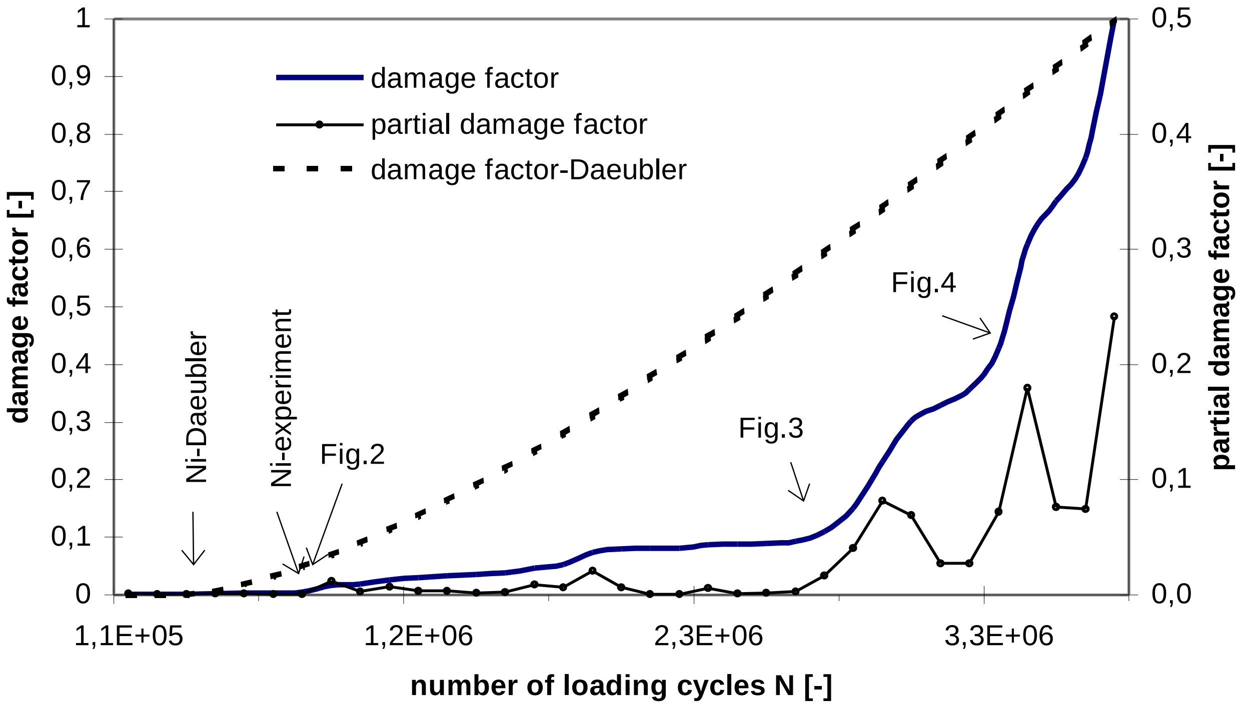

Fig 5

the result of growing deformation amplitude at the tip of a developing crack. This fact establishes a possible conflict with the Daeubler model for corrosion fatigue crack initiation [3]. This model presumes the film rupture events to occur in equidistant time intervals. The actual damage level (amount of dissolved metal) of the tested specimen was estimated from the measurement of CCN transient data. The damage estimation (damage factor $D(N) = \Sigma PD(N_j)$, for $j = 1, \ldots, n_N$, where $PD(N_j)$ is the partial damage factor expressing the damage corresponding to $N_j - N_{j-1} = 108000$ cycles) was calculated by the Faraday law [3], with formal requirement on the value of damage factor to be equal to one at the end of crack initiation process (crack depth of 2 mm). After an incubation period (marked Ni), the damage factor value is increasing like staircase, see Fig. 5. The stairs are shorter and higher with increasing number of cycles. The difference between Daeubler model damage and the experimentally determined damage is significant.

The model presumptions based on a major role of metal matrix plastic deformation (measured on the specimen surface) in rupturing the passive film may be incorrect, too. For example, for steel [6] with higher yield strength Rp0,2 = 639 MPa (cyclic yield strength is estimated [14] to be 607 MPa) and with fatigue limit $S_{ac,\ R=0}$ = 132 MPa for $N_F = 10^7$, corrosion fatigue cracks are initiated due to loading with cycle amplitude $S_{a,\ R=0}$ = 50 MPa, i. e. the amplitude is below 50% of anelasticity limit [15]. These values, i.e. that of the fatigue limit and that of the stress cycle amplitude are corresponding to plastic deformation amplitude values of 10^{-11} and 10^{-16}, respectively. Only reversible motion of dislocation in this loading area is possible, without damage cumulating effect [15]. However, phenomena of both cyclic and static fatigue of ceramics are known [16]. Very thin layers considered in the corrosion fatigue crack initiation analysis have a different behavior with respect to massive ceramics behavior, but for qualitative considerations in a carefully performed evaluation of passive film rupture the knowledge of ceramics fatigue would be applicable.

In spite of the difficulty attempt with current measurement of both the corrosion current noise and acoustic emission was successful. The measurement showed increased acoustic emission activity at some 100 cycles (in order – 100 cycles respond to 0,77 s) before CCN transients occurrence and close to the end of CCN transients. During the dissolution - repassivation event the AE activity is low. Thus, the passive film rupture is a gradual process having duration of 100 cycles in order. In many cases, the AE event coincides with the beginning CCN transient, see Fig. 3. The AE activity increase at the end of the CCN transients reflects a shake - down of new passive film at the crack tip, probably. It is presumed that acoustic emission contributes to the understanding of cycle asymmetry affect, too.

The above mentioned facts, experimental results [6] and [7], and the theoretical analysis [13] suggest the following working hypothesis for the mechanism of film rupture in the initial phase of the corrosion fatigue crack initiation. Repeated ruptures of the passive layer may result from fatigue of the layer in presence of depassivation ions. Presence of depassivation ions in the solution affects parameters of current decay curve (dissoluted metal amount per event), time intervals between subsequent events of passive film rupture and probably even the time interval till the first major passive film rupture (for corrosion fatigue crack initiation).

Decreasing of CCN transients magnitude with crack growing may be explained using results of electrochemical measurements within a simulated corrosion fatigue crack [18]. When the crack tip potential is different from the specimen surface corrosion potential, the potential gradients, steep at the crack tip, reflect the crack geometry, the solution resistivity, and the current flow required to maintain sides of the crack at particular potentials. The IR drop along the crack depth direction is reflected in the current gradients. In the crack mouth, the corrosion current is decreased for deeper crack.

CONCLUSIONS

The fatigue testing resonance - type machine was successfully instrumented for monitoring surface potential, corrosion current noise and acoustic emission during corrosion fatigue test of stainless steel specimens.

During the corrosion fatigue test, there were registered saw shaped transients of corrosion current noise that reflected passive film rupture events on the specimen's surface. The corrosion current noise transients were associated with an increase of the acoustic emission activity.

The results of corrosion current noise measurement show that - for given loading cycle amplitude - the rate of passive film rupture events increasing with the number of loading cycles could be estimated.

In spite of limited experiment scope, the acquired data corrected the ideas concerning the corrosion fatigue crack initiation mechanism. The results of corrosion current noise tests will significantly contribute to the modeling of corrosion fatigue crack initiation for stainless steel under passive electrochemical conditions.

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